




**Figure 3**

(a) The donor–donor (dashed lines) and donor–anion (dotted lines) interactions. (b) The anion ribbon stabilized by N $\cdots$ K contacts. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i)  $-x + 2, -y + 1, -z + 1$ ; (ii)  $-x + 2, -y + 2, -z + 1$ ; (iv)  $x + 1, y + 1, z$ ; (v)  $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (vi)  $x, y + 1, z$ .]

contacts [see Table 1 and Fig. 3(a)], while only one S3 $\cdots$ S3 contact for interdimer interaction pattern *B* is observed. The intra- and inter-dimer interplanar separations within the stack, calculated using the central six atoms of the TTF fragment, are 3.355 (8) and 3.460 (8) Å, respectively.

The complex anion  $\text{K}^+[\text{Hg}(\text{SCN})_4]^{2-}$  is located in a general position near the twofold screw axis  $2_1$ . The Hg atom adopts a tetrahedral coordination involving S atoms, yet each of the SCN ligands adjusts to the crystal environment by rotating around the Hg–S bonds. Note that the conformation of the anion revealed in (I) has not been reported previously. Analysis of structures collected in the Cambridge Structural Database (Bruno *et al.*, 2002; Allen, 2002) indicates that, in the previously reported structures, at most two SCN vectors of the  $[\text{Hg}(\text{SCN})_4]^{2-}$  anion appear to be near collinear instead of three as in the present compound. These three ligands interact with the  $\text{K}^+$  ion *via* short N $\cdots$ K contacts of 2.780 (4)–3.108 (4) Å, stabilizing the anion ribbon as shown in Fig. 3(b). The fourth SCN ligand protrudes out towards the donor

molecules, its terminal N2A atom being involved in a number of hydrogen-bond contacts with the amide groups (Fig. 3a and Table 2). The recurrent tweezer-like hydrogen-bonded seven-membered ring motif (Heuzé *et al.*, 2000; Fourmigué & Batail, 2004) acting upon the combined effect of activated –NH and –Csp<sup>2</sup>H hydrogen-bond donors is again identified here. In addition, the radical cation stacks are connected to adjacent anion ribbons by short O $\cdots$ K [2.606 (3) Å] and S $\cdots$ S contacts along [101]. The coordination polyhedron around the  $\text{K}^+$  cation is a distorted octahedron composed of one O and five N atoms; this differs from the eight- (4N + 4S) and seven-coordination (4N + 3S) of K in  $\alpha$ - and  $\delta$ -(BEDT-TTF)<sub>2</sub>K[Hg(SCN)<sub>4</sub>], respectively (Oshima *et al.*, 1989; Kazheva *et al.*, 2002). The crystals of (I) are semiconducting, as expected for a dimerized stack.

## Experimental

Single crystals of (I) were obtained by electrooxidation, at constant current ( $I = 2 \mu\text{A}$ ) and 293 K, of EDT-TTF-CONHMe in 1,1,2-trichloroethane solution containing 10% (v/v) dried ethanol. Plate-like crystals grow on the anode after three weeks.

### Crystal data

$(\text{C}_{10}\text{H}_9\text{NOS}_6)\text{K}[\text{Hg}(\text{SCN})_4]$	$V = 2649.2 (6) \text{ \AA}^3$
$M_r = 823.55$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 13.7129 (17) \text{ \AA}$	$\mu = 6.78 \text{ mm}^{-1}$
$b = 7.9403 (9) \text{ \AA}$	$T = 294 (2) \text{ K}$
$c = 24.862 (3) \text{ \AA}$	$0.15 \times 0.06 \times 0.02 \text{ mm}$
$\beta = 101.879 (5)^\circ$	

### Data collection

Bruker X8 APEX CCD diffractometer	25746 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	6827 independent reflections
$T_{\min} = 0.430, T_{\max} = 0.876$	4594 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.040$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	309 parameters
$wR(F^2) = 0.069$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{\max} = 0.88 \text{ e \AA}^{-3}$
6827 reflections	$\Delta\rho_{\min} = -0.61 \text{ e \AA}^{-3}$

**Table 1**

Intermolecular contacts (Å) in the donor stack.

S1 $\cdots$ S4 <sup>i</sup>	3.384 (1)	C1 $\cdots$ C1 <sup>i</sup>	3.577 (7)
S2 $\cdots$ S3 <sup>i</sup>	3.350 (1)	C4 $\cdots$ C5 <sup>i</sup>	3.394 (5)
S3 $\cdots$ S3 <sup>ii</sup>	3.574 (2)	C1 $\cdots$ C2 <sup>i</sup>	3.363 (5)
S3 $\cdots$ C4 <sup>i</sup>	3.620 (3)	C3 $\cdots$ C6 <sup>i</sup>	3.442 (5)
S5 $\cdots$ C9 <sup>i</sup>	3.543 (4)	C3 $\cdots$ C5 <sup>i</sup>	3.564 (5)

Symmetry codes: (i)  $-x + 2, -y + 1, -z + 1$ ; (ii)  $-x + 2, -y + 2, -z + 1$ .

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7A–H7C $\cdots$ N1 <sup>i</sup>	0.97	2.63	3.45 (2)	142
C8–H8B $\cdots$ N2A <sup>iii</sup>	0.97	2.71	3.539 (9)	144
N1–H1 $\cdots$ N2A <sup>iv</sup>	0.86	2.10	2.938 (5)	165
C5–H5 $\cdots$ N2A <sup>iv</sup>	0.93	2.41	3.298 (5)	160

Symmetry codes: (i)  $-x + 2, -y + 1, -z + 1$ ; (iii)  $-x + 1, -y, -z + 1$ ; (iv)  $x + 1, y + 1, z$ .

The final refinement was carried out in the space group  $P2_1/n$ , chosen on the basis of the systematic absences and successful refinement of the structure. The positions of the methyl and ethylene H atoms were calculated as riding, in accordance with the HFIX 137 and HFIX 23 instructions of *SHELXL97* (Sheldrick, 1997), with  $U_{\text{iso}}(\text{H})$  values of 1.5 and 1.2 times  $U_{\text{eq}}(\text{C})$ , respectively. The H atoms of  $-\text{Csp}^2\text{H}$  and  $-\text{NH}$  groups were located in difference electron-density syntheses and constrained by an AFIX 43 instruction [ $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ ]. The terminal ethylene group of the radical cation is disordered over two positions in an approximate 3:1 ratio and C–C distances were restrained to be equal.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2* and *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV3066). Services for accessing these data are described at the back of the journal.

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